

Institute of Paper Science and Technology
Central Office

SEGREGATION OF WOOD CHIP/BARK MIXTURES USING LIQUID FLOTATION PROCEDURES

Project 2977

Report Three

A Progress Report

to

MEMBERS OF GROUP PROJECT 2977

June 1, 1972

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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Appleton, Wisconsin

SEGREGATION OF WOOD CHIP/BARK MIXTURES
USING LIQUID FLOTATION PROCEDURES

SUMMARY

The third quarter of Project 2977 involved investigations into the flotation behavior of loblolly pine, shagbark hickory, and white spruce and included investigations of various flotation system modifications meant to improve the segregation of wood chips from bark chips. The tree species were described and pure fractions of wood chips and bark chips were tested to determine the influence of moisture content, chip size, dwell time in the flotation medium, and type of wood (core or slab) on the flotation behavior of the wood or bark.

The expected held true for all species. Small chips sank faster than large chips and wet chips sank faster than the dry chips but it was here that the general similarities ended. For loblolly pine, the bark tended to float and the wood tended to sink. Complete submersion of wood required 144 hours, at which time a high proportion of bark was still floating. Corewood tended to float longer than mature wood. Accelerated segregation, wood sinking and bark floating, was performed in 15-20 minutes using 45% moisture content chips and a simple pressure cooker system. A high percentage (93-99%) of wood was recovered with a minimum amount of bark. Mixtures of corewood and slabwood which simulated pine from natural stands and plantations were evaluated. The results indicated a slight difference could be anticipated with better wood recovery from natural stands of pine.

The shagbark hickory investigation revealed successful water flotation segregation of wood from bark chips can be obtained in under 30 minutes using 20%

moisture content chips. The bark sank more quickly than the wood allowing recovery of wood as floating chips.

For the white spruce, the flotation behavior of the bark and wood was too similar to allow good segregation. In the initial tests the best segregation took place at 48-72 hours with 20% moisture content chips. From 79-96% of the wood was recovered with 0.3-2.2% bark in the wood. Modifications applied to the flotation system included steam pressure, higher water temperature, compression, and moisture content changes. Using chips at 35% moisture content and compression reduced the time for the best segregation to 30 hours, but decreased wood recovery and increased contamination.

INTRODUCTION

This report marks the completion of the third quarter of work for Project 2977, "Segregation of Wood Chip/Bark Mixtures Using Liquid Flotation Procedures." During the first half of the project, five species of hardwoods were investigated. Workable segregation systems were developed for quaking aspen, bur oak, sugar maple, and white birch while one species, eastern cottonwood, was found difficult to segregate via liquid flotation.

Work during the past quarter was concentrated on loblolly pine, shagbark hickory, and white spruce. The work covered the basic flotation tests, which determined flotation characteristics, and modified flotation tests, which investigated system changes likely to bring about a successful liquid flotation segregation of bark from wood chips. The results of the work completed this past quarter are reported by tree species.

METHODS AND MATERIALS

In the initial stages of this project some arbitrary limits were set regarding objectives acceptable for segregation of wood chips from bark. "Depending on the mill, the process, and the end product, the acceptable level of bark contamination lies somewhere between virtually no bark in the wood to as high as 10%. For a practicable approach to the problem it was decided that 1 to 3% was a reasonable bark contamination percentage, and further reduction of bark would be considered a problem specific to the particular mill." In addition, it was decided that 10% is the maximum acceptable allowance for wood loss.

The basic flotation test used with the first five species was designed to isolate and describe, as far as practical, the several factors influencing chip flotation of the given species. The two factors chosen at the beginning of the project were chip size and moisture content. Pure samples of wood or bark were used and a compression step that worked well with aspen was included as part of the standard test. The floating chips, after the initial five-minute agitated flotation, were compressed and the agitated flotation was extended five more minutes at which time the tests were terminated and the oven-dry weight of the sunken and floating chips determined.

After the work on the first five species was completed, the basic flotation tests were reevaluated and it was decided that certain changes should be made. The compression step often added little to the overall flotation behavior data and it sometimes complicated the basic test but was a valuable inclusion in the testing of modified systems. Experience in the early testing indicated that dwell time of the chips in water could have an important bearing on the development of a flotation segregation system for a species. The compression

step was therefore eliminated from the basic flotation test and "dwell" added by extending the flotation up to a maximum of 168 hours.

The new species being evaluated presented an additional problem in that it appeared there was considerable variation from the center of the tree outward in wood properties that might influence flotation results. With this in mind the "juvenile" wood of the conifers (first eight annual rings) and the "heartwood" of hickory was separated from the mature wood by a slabbing procedure. The two fractions, "corewood" and "slabwood," were chipped and evaluated separately. Several flotation runs were also made employing mixtures of the two fractions and using a procedure which involved dye marking one fraction for easy identification.

The modified flotation tests were set up for each species as the information from the basic flotation tests most logically indicated. No modifications were necessary for the shagbark hickory, steam pressure treatments were needed for the loblolly pine, and both steam pressure and compression treatments were tested for the white spruce.

SAMPLE COLLECTION AND PREPARATION

The three tree species tested in this quarter were:

Loblolly pine (Pinus taeda L.)

Shagbark hickory [Carya oviata (Mill.) K. Koch]

White spruce [Picea glauca (Moench) Voss]

Two trees each of plantation-grown and old-field loblolly pine¹ were furnished in November by Olinkraft, Inc. from their land near West Monroe, Louisiana. Two trees each of Wisconsin-grown shagbark hickory and plantation white spruce were cut for use in the flotation tests in November. The description of the trees used are given in Table I.

TABLE I
SAMPLE TREE DESCRIPTIONS

Material	Age, years	Height, ft.	Diameter at 4.5 ft., inches	Av. Sp. Gr. ^a				Corewood, % by vol.	Bark, % by o.d. wt.
				-----Wood-----					
				Core	Slab	Total	Bark		
Loblolly pine	16 ^b	44	7.3	0.450	0.510	0.477	0.223	48.0	8.3
	16 ^b	46	7.0	0.400	0.470	0.441	0.234	44.6	9.9
	22 ^c	59	7.6	0.440	0.545	0.504	0.238	25.6	8.0
	22 ^c	53	7.1	0.410	0.480	0.455	0.232	33.5	8.5
Shagbark hickory	100±	47	10.2	0.650	0.510	0.621	0.601	69.7	16.3
	100±	50	9.8	0.630	0.510	0.591	0.594	68.2	14.4
White spruce	27	41	6.7	0.290	0.320	0.316	0.195	29.6	6.3
	27	36	7.2	0.310	0.320	0.325	0.218	26.6	7.1

^aBased on oven-dry weight (g.)/green volume (cc.).

^bPlantation grown.

^cOld-field pine.

Sample disks were taken at four-foot intervals up the tree for specific gravity, percentage bark, and percentage corewood determinations. Specific gravity was determined using TAPPI "on the balance" method T 18 m-53. Corewood was considered to be juvenile wood (first eight annual rings) for the loblolly pine and white spruce and heartwood for the shagbark hickory.

¹Old-field pine is used throughout this report to designate natural stands that have seeded in on old-field sites.

The specific gravities of the various woods in general are in agreement with values found in the literature (Appendix Table X). The plantation-grown white spruce has slightly lower wood and bark specific gravity values than reported for natural stands of mature white spruce. As was expected, the old-field loblolly pine had higher specific gravity than the plantation-grown loblolly pine due to a smaller proportion of corewood in the old-field pine.

The core wood of each of the four-foot bolts for each species was marked and a carriage saw used to cut the slabwood from the corewood as best as possible. The slabs were tied in bundles and chipped by species. The corewood was also chipped separately by species. The chipper used was a 41-inch, 4-knife machine made by Carthage Machine Company, and the newly sharpened knives were set to deliver chips at a nominal 3/4-inch length.

After chipping the slabwood and the corewood, chips of each species were screened on a 24-inch Sweco vibratory screen fitted with 3/4, 1/2, and 1/4-inch mesh screens. The chips were charged to the top (3/4-inch mesh) screen where the obviously oversized materials were picked off manually. The screen delivered the sized materials continuously so that 4 screenings were recovered, i.e., (1) on 3/4-inch, (2) through 3/4-inch and on 1/2-inch mesh, (3) through 1/2 and on 1/4-inch mesh, and (4) through 1/4-inch mesh. The data concerning this preliminary work are given in Table II. The differences in the proportions of the various sizes of chips appeared to be related to the resistance of the wood to the impact of the chipper blades.

The fresh weights of each size class and type of wood were determined and representative samples taken to determine moisture content. In addition, representative fractions of "on 1/4-inch" and "on 3/4-inch" chips were hand

TABLE II
CHIP DESCRIPTIONS

Chip Size	Composition, % by fresh wt.			Moisture Content at Chipping, (fr.wt.-o.d. wt./fr.wt.), %			Bark, % by dry wt.	
	Core	Slab	Comb.	Core	Slab	Bark	Of Size Class	Of Total Bark
<u>Loblolly Pine</u>								
Oversize	0.5	6.2	3.4	--	--	--	--	--
On 3/4-inch	8.6	33.1	21.0	51.0	43.4	36.6	9	26
On 1/2-inch	66.0	42.1	53.9	51.3	-----47.0-----		7	41
On 1/4-inch	20.6	16.0	18.3	52.0	42.6	35.8	17	33
Fines	4.3	2.6	3.4	--	--	--	--	--
<u>Shagbark Hickory</u>								
Oversize	2.0	9.9	4.3	--	--	--	--	--
On 3/4-inch	35.5	38.2	36.3	34.5	31.4	26.1	16	40
On 1/2-inch	53.0	34.6	47.6	35.4	-----29.4-----		11	37
On 1/4-inch	8.5	13.6	10.0	34.2	29.1	19.0	32	23
Fines	1.0	3.7	1.8	--	--	--	--	--
<u>White Spruce</u>								
Oversize	4.1	5.9	5.3	--	--	--	--	--
On 3/4-inch	24.8	47.5	39.0	46.2	58.0	62.5	25	78
On 1/2-inch	52.9	34.7	41.5	64.2	-----60.7-----		6	19
On 1/4-inch	14.9	9.9	11.8	57.8	58.7	52.8	4	3
Fines	3.3	2.0	2.4	--	--	--	--	--

sorted into wood or bark chips from the slab chips. There were then pure samples of corewood, slabwood, and bark of "on 1/4-inch" and "on 3/4-inch" available for testing.

EXPERIMENTAL PROCEDURE

For simplicity in interpreting results and ease in making comparisons between species, the testing has been divided into "basic tests" and "modified tests." The basic flotation test used in the first half of this project and the reasons for altering the tests were described earlier in the report. The specifics of the new basic flotation tests and the general descriptions of modified systems are described in the two subsections following.

Basic Flotation Testing Procedure

This test begins with a five-minute agitated flotation, which is followed by a flotation involving intermittent agitation and water changes. The flotation can be extended, if necessary, to a maximum 7-day period. Sunken chips were removed at 5 minutes, 1/4, 1/2, 1, 4, 6, and 24 hours and at 24-hour intervals thereafter until the test was terminated. Bark and wood chips of "on 1/4-inch" (small) and "on 3/4-inch" (large) size were tested at 20 and 45% moisture contents. In addition, both corewood and slabwood chips of the loblolly pine were tested. The spruce corewood and shagbark hickory sapwood (slab) chips were marked by mixing each with a small amount of powdered dye. The flotation tests were run on an appropriate mixture of corewood and slabwood.

All tests were run in duplicate. The samples for the bark tests were 100 grams oven-dry weight, while the wood samples were 200 grams oven-dry weight. Sample chips were adjusted to the proper moisture content either by adding distilled water or by drying the chips. The adjusted moisture contents were checked by

dividing the total oven-dry weight of the sample, determined after the flotation, by the fresh weight of the sample before the flotation. With the exception of a few instances stated later, the adjusted moisture contents fall within $\pm 4\%$ of the intended percent.

The flotation was initiated in the clear acrylic vessel described in Progress Report One. The vessel was filled with 20 liters of city tap water and the temperature adjusted to 22°C. The surface area of the water was 560 sq.cm. After the chip sample was placed in the vessel, the top was closed with a device which kept the chips submerged at least 6 cm. below the water surface. A hand-operated 4-blade paddle agitated the chips for the first five minutes. At the end of this time the top was removed and the floaters skimmed off and placed in a glass vessel of a diameter similar to the acrylic vessel but filled with about 6.5 liters of water. The remainder of the flotation took place in the glass vessel with sinkers taken out at the appropriate intervals. The recovered chips were air dried overnight and then oven dried 24 hours and their oven-dry weight determined.

The data were compiled for the various flotations, the averages for the duplicated runs determined and these values listed in tables by species. This information is included and used in later sections of this report when discussing the reaction to flotation of the wood and bark chips for each of the three species.

As was stated earlier, "pure bark" and "pure wood" samples were tested to facilitate the description of the process and the interpretation of the data. A complete study must take into account how a wood-bark chip mixture might behave. To accomplish this, the data available on pure fractions were used to interpret, by means of a mathematical formula, the percentage of bark that would remain as

"contamination" in the recovered wood. The term used to describe the results of this calculation is "bark contamination factor" (BCF).

The amount of wood recovered is listed as "accumulated wood recovery" (AWR). This figure represents the total percent of wood that can be recovered in a given time interval. Whether the wood is sunken or floating is indicated and the time interval is assumed to be from the start to the time stated, unless otherwise designated.

The BCF is the assumed amount of bark in the material recovered from a theoretical wood-bark mixture expressed as a percentage of the oven-dry weight of the recovered material (wood and bark). For the purposes of this report, a theoretical mixture of 25% bark is used. The ratio is quite arbitrary and is in fact high for most species.

Modified Flotation Testing Procedure

If a species did not show reasonable segregation, 90% or more wood recovery with less than 3% bark contaminating the wood, the flotation behavior was studied and a decision made as to what modifications might give reasonable segregation. The modifications were then tested with preliminary runs on wood-bark mixtures of "on 1/2-inch" chips. After the preliminary runs, the best previously tested modification was then run on pure fractions of "on 1/4-inch" and "on 3/4-inch" wood and bark chips.

Since the modifications varied from species to species, the procedures for the test are not described here but are described in the results section of each species. Modifications considered were: water temperature, chemical additions, atmospheric pressure changes, compression, moisture content changes, sequence variation, and mechanical manipulation of chips.

The two principal pieces of apparatus used for the modified tests in this report period were a 10-gallon resin kettle and a mechanically-driven glue spreader. The resin kettle is fitted with a pressure top and a steam jacket and can be pressurized directly or heated to create pressure indirectly. For the purposes of the modified tests, the kettle was partially filled with heated water, the chips submerged in the water, and the kettle pressurized directly with live steam. The resin kettle will be referred to as a "pressure cooker," a better recognized term, for the remainder of the report.

The glue spreader was used simply as a source of spring-loaded rolls that were used to compress the chips. It has two rolls, one steel idler roll opposed by a steel chain-driven roll covered with a thin, grooved rubber cover.

RESULTS OF FLOTATION EXPERIMENTS

LOBLOLLY PINE BASIC FLOTATION TESTS

As was stated earlier, the loblolly pine work tested three kinds of chips, corewood (first 8 annual rings), slabwood (rings beyond year 8) and bark. Using these three kinds of chips, the source of wood, i.e., plantation or old-field, can be disregarded during the test. Since the basic difference between sources is the amount of corewood, the source can be best handled by computing the proper ratio of core to mature wood and adjusting the AWR (accumulated wood recovery) and the BCF (bark contamination factor) accordingly. Each kind of chip (core, slab, and bark) was tested for its flotation behavior using small ("on 1/4-inch") and large ("on 3/4-inch") chips at two moisture contents, 20 and 45%.

The results of the basic flotation tests, listed in Table III, show a tendency for the wood to sink and the bark to float. The slabwood consistently sank faster than the corewood. The 45% moisture content wood chips sank faster than the 20% moisture content chips but there was little difference in behavior of the bark at the different moisture contents. The small wood chips sank faster than the large ones and more of the small bark chips sank than the large ones. The large slabwood chips sank more readily than the other wood chips and this could be attributed to the fact that the actual moisture content was 54%, 9% higher than the other chips.

Previous work at The Institute of Paper Chemistry with merchantable-size loblolly pine indicated corewood could range from 9 to 53%. Realizing a great variation can exist, for the purposes of this report the average percent corewood of the trees supplied was used to determine the variation in wood recovery

TABLE III
BASIC FLOTATION TESTS
LOBLOLLY PINE^a

Sunken Chips Taken Off At:	On 1/4-Inch Chips					On 3/4-Inch Chips				
	Sinks, %		AWR ^b		BCF, %	Sinks, %		AWR ^b		BCF, %
	Wood	Bark	Plantation	Old		Wood	Bark	Plantation	Old	
	Core	Mature	Field	Field		Core	Mature	Field	Field	
	<u>20% Moisture Content</u>									
5 Min.	5.6	16.4	4.4			0	4.6	5.1		
15 Min.	0.2	0	0			0	0	0		
30 Min.	0.6	0.4	0			0	0	0		
1 Hour	0.5	1.3	0			0	0	0		
4 Hours	7.6	10.8	0.6			4.7	5.6	0.6		
6 Hours	2.9	2.6	0			0	2.4	0		
24 Hours	34.8	31.8	1.2			45.4	45.1	1.2		
48 Hours	29.4	25.8	1.4			30.7	26.7	1.0		
72 Hours	12.4	5.2	1.0			11.6	11.4	0		
96 Hours	4.2	4.2	2.0	98.4	3.5	0	2.6	0.9	95.7	3.0
120 Hours	1.2	0.7	5.2	99.3	5.0	6.8	0	0.6	98.6	3.1
144 Hours	0.3	0.6	0	99.8	5.0	0.8	1.3	0.5	99.8	3.2
Floater	0.3	0.2	84.2			0	0.3	90.1		
	<u>45% Moisture Content</u>									
5 Min.	26.7	56.4	4.7			29.1	95.2	2.0		
15 Min.	0.5	<0.1	0			0	0	0		
30 Min.	0.3	0	0			0.9	0	0		
1 Hour	0.6	1.3	0			2.3	0	0		
2 Hours	6.1	7.5	0.7			14.9	0	0		
6 Hours	1.6	2.3	0			0	2.2	0		
24 Hours	27.2	22.3	1.3	77.3	2.8	27.8	2.6	2.6	88.4	1.7
48 Hours	22.3	6.9	1.2	91.7	2.8	19.0	0	0	97.2	1.6
72 Hours	11.5	1.8	2.0	97.9	3.3	3.9	0	1.9	98.3	1.5
96 Hours	1.8	0.7	2.0			1.7	0	0		
120 Hours	0.8	0.5	2.7			0	0	0.1		
144 Hours	0.4	<0.1	17.0			0.4	0	1.4		
Floater	0.2	0.2	68.4			0	0	93.0		

^aData in table are averages of duplicate determinations and are percentages based on oven-dry weights. Corewood is considered to be the first eight annual rings and mature wood is rings nine and beyond. The plantation wood is considered to have 46.3% corewood while the old-field pine has 29.6%.

^bAWR = Accumulated Wood Recovery; wood recovered from the start of the flotation to the time indicated.

^cBCF is simply a calculation of % bark by oven-dry weight included with the recovered wood from a supposed original mixture of 75% wood and 25% bark.

^dOn 3/4-inch chips of mature wood, 45% moisture content were actually 54% moisture content.

due to wood source. Plantation-grown wood is considered to have 46.3% corewood and the old-field pine to have 29.6% corewood. The accumulated wood recovery (AWR) is figured as: accumulated wood recovery percent = (ratio of corewood x percent corewood recovered) + (ratio of slabwood x percent slabwood recovered). The bark contamination factor (BCF), assuming 25% bark in the original sample, can then be computed simply as: $BCF \% = [\% \text{ bark recovered} / (\% \text{ bark recovered} + 3 (\text{AWR} \%))]]$.

The BCF and AWR figures in Table III show wood-bark chip segregation via liquid flotation is obtainable for either plantation or old-field grown loblolly pine but only after a fairly long (72 hours) flotation time. For both the old-field and the plantation-grown pine, more than 90% of the wood was recovered as sinking material with less than 2.8% bark contamination (BCF) at 48 hours.

LOBLOLLY PINE MODIFIED FLOTATION TESTS

It was evident from the basic flotation data that wood-bark chip segregation via liquid flotation is possible with loblolly pine. The bark tends to float regardless of moisture contents tested and the wood sinks more quickly with increased moisture content. Both factors indicate that loblolly wood-bark chips can probably be best processed fresh, with little or no drying. The problem to work on was shortening the time required to affect a "reasonable" segregation. The best modification, of those tried with the initial five species, appeared to be the steam-pressure treatment.

The steam-pressure treatment was tried with "on 1/2-inch" chips and the results of duplicate determinations indicated an average of 99.2% wood was recovered with BCF values of 0.4 and 2.6%. The bark sinking and contaminating the

recovered wood was mostly the slippery inner bark and probably is not a problem to most pulping processes. The conditions of the test are listed in Fig. 1.

The flotation system was repeated with pure fractions of corewood, slabwood and bark. Duplicate tests were made on each class with the two types of wood and the bark being treated in the pressure cooker together, each in a separate container.

The results, listed in Table IV, show a good segregation for the large chips and a less than optimum segregation for the smaller chips (Fig. 2). For both plantation and old-field grown loblolly pine, computations show over 99% of the small wood chips were recovered with 3.8% bark contamination and 93% or more of the large chips were recovered with 2.9% bark contamination. A large percentage of the sunken bark was the inner bark in both size classes. It is felt that less bark would be included in the small chips if a shorter treatment time was used. However, some loss in recovered wood could be expected.

The results of the first flotation, before the pressure cooker treatment, are interesting in that a high percentage of wood chips sank at that point. The fact that a greater amount of the "small" corewood chips sank in the first flotation, more than sank in the basic flotation test, can be attributed to an actual moisture content 7% higher (52%) than in the basic flotation test. This along with the results of the basic flotation tests, where the large mature chips had an actual moisture content of 54% instead of the intended 45% and sank more rapidly, further substantiates the possibility of a water flotation segregation of loblolly pine wood-bark chips at moisture contents of 50-55%.

FLOTATION FLOW CHART
LOBLOLLY PINE

PURE FRACTION UNDER STEAM PRESSURE
45% MOISTURE CONTENT - DUPLICATE RUNS
1/4" & 3/4" CHIPS OF EACH - COREWOOD, SLABWOOD, BARK
WATER AT 85°C. IN STEAM KETTLE; ALL OTHER WATER AT 22°C.

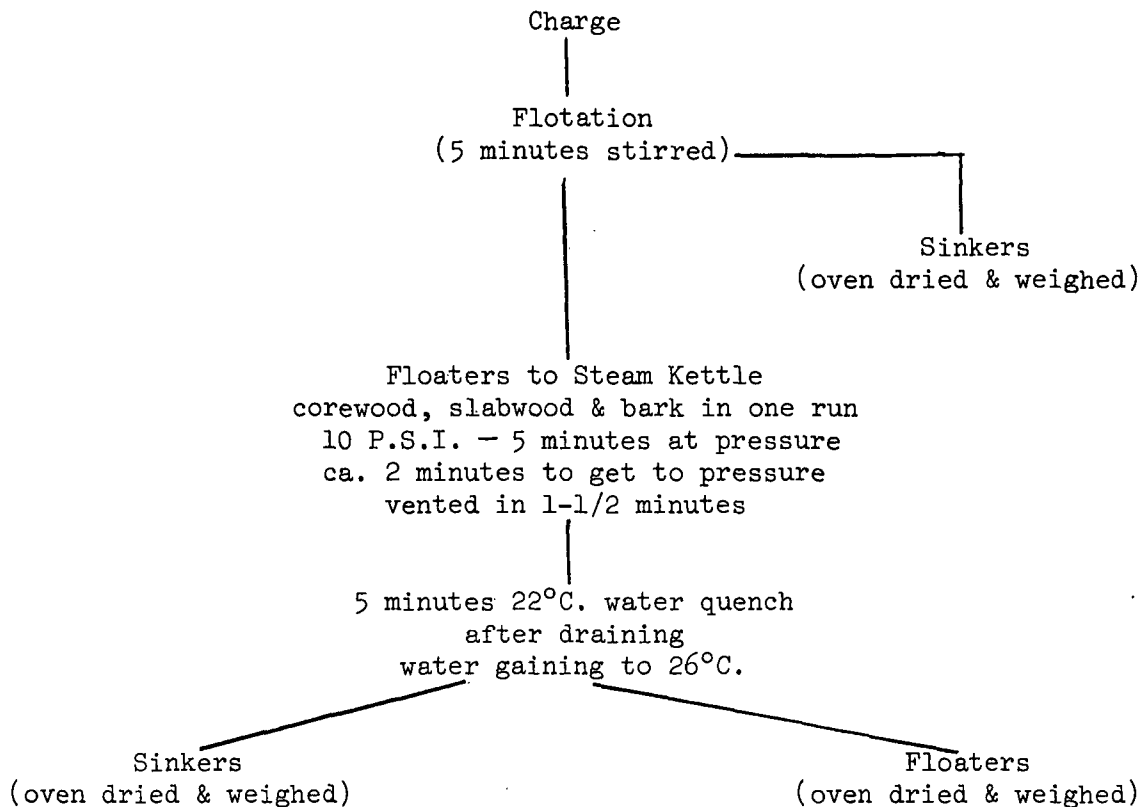


Figure 1. Schematic Diagram for the Modified Flotation Procedure Used Successfully on Loblolly Pine Wood-Bark Chips

TABLE IV
LOBLOLLY PINE
45% MOISTURE CONTENT^a

Sunken Chips Taken Off After:	On 1/4-Inch Chips				On 3/4-Inch Chips			
	Sinkers, %		BCF, % ^c		Sinkers, %		BCF, % ^c	
	AWR ^b		AWR ^b		AWR ^b		AWR ^b	
	Wood	Core ^d	Plantation	Field	Wood	Core	Plantation	Field
1st Flotation	62.6	49.6	5.2		26.8	30.5	4.4	
Steam pressure treatment & 2nd flotation	37.2	50.1	6.7	99.7	59.2	68.6	3.9	95.2
Floating chips	0.2	0.3	88.1		14.0	0.9	91.7	

^aData in table are averages of duplicate determinations and are percentages based on oven-dry weights. Corewood is considered to be the first eight annual rings and mature wood is rings nine and beyond. The plantation wood is considered to have 46.3% corewood while the old-field pine has 29.6%. Tests utilized a pressure cooker treatment. Conditions: 1st flotation is a 5-minute flotation in 22°C. water; the steam pressure treatment consisted of placing the chips in 85°C. water in a pressure cooker, driving the pressure to 10 p.s.i. and holding for 5 minutes, total time in bath about 8 minutes, 2nd flotation then performed by floating chips in 22°C. water for 5 minutes.

^bAWR = Accumulated Wood Recovery; wood recovered from the start of the flotation to the time indicated.

^cBCF is simply a calculation of % bark by oven-dry weight included with the recovered wood from a supposed original mixture of 75% wood and 25% bark.

^dActual moisture content of the on 1/4-inch core chips was 52% instead of the intended 45%.

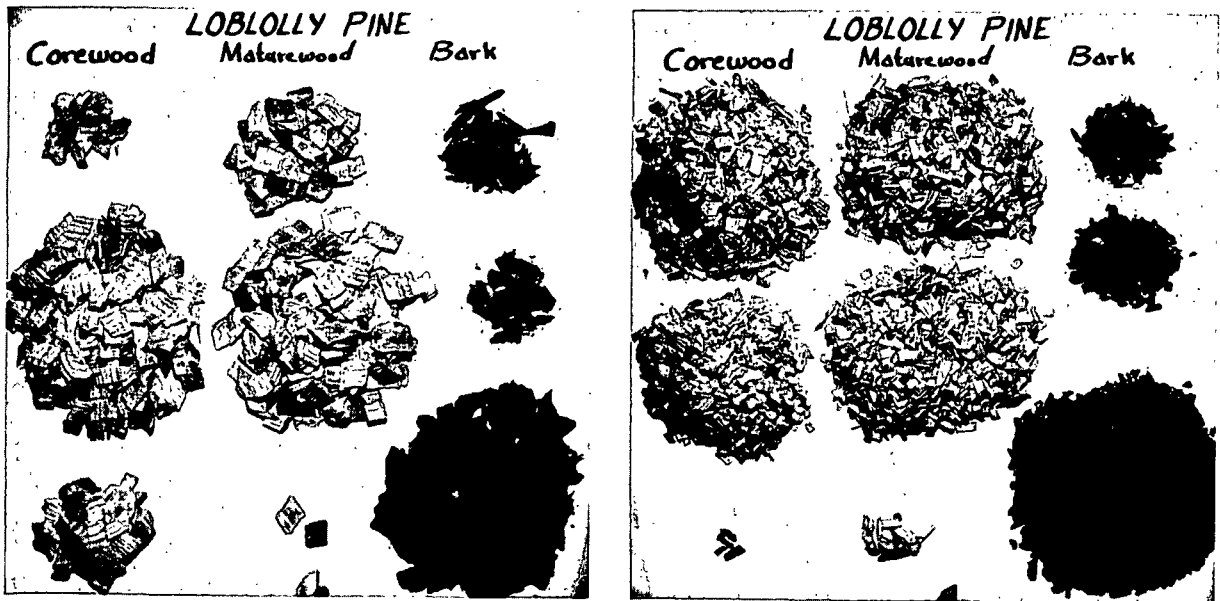


Figure 2. Pictured Above are the Results of One of the Steam-Pressure Modified Flotation Tests on Loblolly Pine (Small Chips Right, Large Chips Left). From the top Down are Chips Sinking After First Flotation, Chips Sinking in Flotation After Steam-Pressure Treatment, and Those Floating

SHAGBARK HICKORY BASIC FLOTATION TESTS

The slabwood (sapwood) of the shagbark hickory was marked with powdered dye and processed through the basic flotation tests with enough corewood (heartwood) to simulate the proportion of the two woods in the original bolts. If differences in flotation characteristics arose between the two woods, it could be easily observed because of the marked chips.

The results of the test are presented in Table V. As can be seen from the data, both bark and wood sink quite readily at a high (45%) moisture content. A differentiation in flotation characteristics between the bark and wood chips was observed when 20% moisture content chips were processed. The wood tended to float

TABLE V
BASIC FLOTATION TESTS
SHAGBARK HICKORY^a

Sunken Chips Taken Off At:	On 1/4-Inch Chips				On 3/4-Inch Chips			
	Sinkers, %		AWR ^b Recovery Floaters, %		Sinkers, %		AWR ^b Recovery Floaters, %	
	Wood	Bark		BCF, % ^c	Wood	Bark		BCF, % ^c
<u>20% Moisture Content</u>								
5 Min.	6.3	96.6	93.7	1.2	3.1	68.8	96.9	9.7
15 Min.	1.3	1.2	92.4	0.8	0.7	17.8	96.2	4.4
30 Min.	3.0	0	89.4	0.8	2.4	4.7	93.8	3.0
1 Hour	13.0	0.9			1.3	8.7		
4 Hours	42.0	0.7			26.2	0		
6 Hours	4.6	0			10.2	0		
24 Hours	20.9	0.5			39.6	0		
48 Hours	7.8	0			9.3	0		
72 Hours	0.8	0			1.4	0		
Floaters	0.8	0.1			5.8	0		
<u>45% Moisture Content</u>								
5 Min.	98.7	100.0	1.3	0	91.0	100.0	9.0	0
15 Min.	0	0			0.6	0		
30 Min.	0	0			0	0		
1 Hour	0	0			0.9	0		
4 Hours	0.2	0			1.8	0		
6 Hours	0.2	0			0.2	0		
24 Hours	0.8	0			4.2	0		
48 Hours	0.1	0			0.3	0		
72 Hours	0	0			0	0		
Floaters	<0.1	0			1.0	0		

^aData in table are averages of duplicate determinations and are percentages based on oven-dry weights.

^bAWR = Accumulated Wood Recovery; wood recovered from the start of the flotation to the time indicated.

^cBCF is simply a calculation of % bark by oven-dry weight included with the recovered wood from a supposed original mixture of 75% wood and 25% bark.

longer than the bark and the smaller chips of both bark and wood sank more readily than the larger ones. As can be seen from the AWR (accumulated wood recovery percent) and the BCF (bark contamination factor) values in the table, 90% or more of the wood was recovered with 3.0% or less bark at 15 minutes for the small chips and at 30 minutes for the larger chips (Fig. 3). Since this is at or within the limits set, it was assumed a satisfactory segregation of shagbark hickory bark-wood chips could result at 20% moisture content with the simple basic flotation system. No further tests were performed on the hickory.

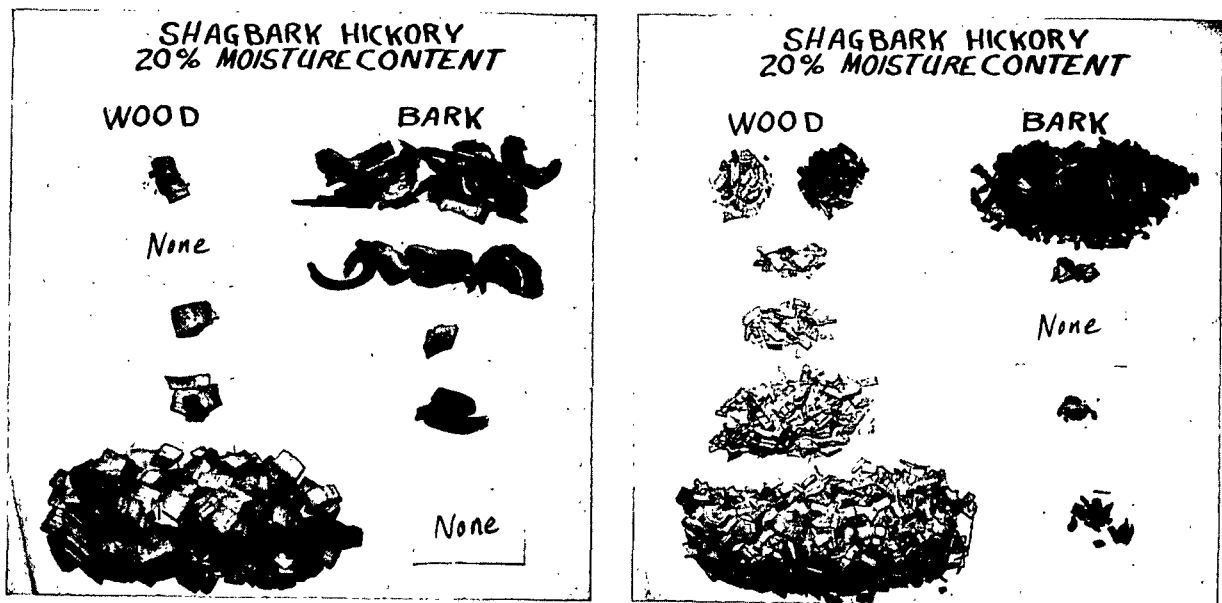


Figure 3. Results of the Basic Flotation Test with Shagbark Hickory Chips (Large Left and Small Right). Shown from Top to Bottom are the Sinkers from 5 Minutes, 15 Minutes, 30 Minutes, and 1 Hour and the Floaters After 1 Hour are on the Bottom

WHITE SPRUCE BASIC FLOTATION TEST

The white spruce chips were handled much like the hickory except the corewood instead of the slabwood was marked with powdered dye. The results of the basic flotation tests are listed in Table VI. As can be seen from the data, the bark was more inclined to sink than the wood. The difference was not great enough to allow a clear-cut segregation of wood from bark using the basic system. The one exception was the large chips (on 3/4-inch) at 20% moisture content. Some difficulty was experienced in obtaining the proper moisture contents of the bark, due in part to a short supply of bark. Because of this, the actual moisture contents are listed in the table. It did not appear that reruns at the proper moisture content would have improved the picture, so the data were used as originally obtained.

In general, for both bark and wood, the smaller chips sank faster than the larger chips and the wetter (45% moisture content) chips sank sooner than the drier (20% moisture content) chips. While both core and slabwood chips sank at most time intervals, in general, the mature wood sank sooner and, as a result, a majority of the chips floating after 72 hours were juvenile wood.

WHITE SPRUCE MODIFIED FLOTATION TESTS

Since the results of the basic flotation test indicated an adequate wood-bark chip segregation could not be obtained for white spruce using the basic flotation system, several modifications were tried. Basic flotation test data indicated that the maximum wood recovery with the minimum amount of bark contamination occurred in the wood with 20% moisture content. The exact time of this occurrence varied from 24 hours for the small chips to 72 hours for the large chips. The fact that 96.5% of the large chips were recovered at 72 hours with a

TABLE VI
BASIC FLOTATION TEST
WHITE SPRUCE^a

Sunken Chips Taken Off At:	On 1/4-Inch Chips				On 3/4-Inch Chips			
	Sinkers, %		AWR ^b Recovery Floaters, %		Sinkers, %		AWR ^b Recovery Floaters, %	
	Wood	Bark		BCF, % ^c	Wood	Bark		BCF, %
Intended 20% Moisture Content ^d								
	21%	27%	Actual Moisture Content		24%	37%		
5 Min.	0	11.4			0	0.8		
15 Min.	0	0			0	0		
30 Min.	0.6	0.7			0	0		
1 Hour	0	2.0			0	0.4		
2 Hours	1.2	7.3			0	2.0		
4 Hours	0.8	13.7			0	2.8		
6 Hours	0	8.4			0	0		
24 Hours	5.2	41.4	92.2	5.3	0	26.9		
48 Hours	13.2	9.7	79.0	2.2	1.9	55.6	98.1	3.8
72 Hours	24.0	2.7			1.6	10.7	96.5	0.3
96 Hours	27.9	>0			3.9	0		
120 Hours	14.1	>0			11.0	0		
144 Hours	6.4	>0			11.4	0		
168 Hours	2.4	2.7			18.2	0.6		
Floaters	4.2	0			52.0	0.2		
Intended 45% Moisture Content ^d								
	45%	58%	Actual Moisture Content		48%	47%		
5 Min.	6.2	7.7			1.1	1.4		
15 Min.	0	0			0	0		
30 Min.	0	0.8			0	0.2		
1 Hour	0.4	1.9			0	0		
2 Hours	0.4	4.7			0.4	0.8		
4 Hours	1.1	7.6			0	1.2		
6 Hours	0	7.0	91.9	13.3	0	0		
24 Hours	13.8	35.0	78.1	13.1	7.0	29.4	91.5	19.6
48 Hours	20.2	27.5			10.8	45.0	80.7	8.3
72 Hours	27.4	2.8			13.7	21.3		
96 Hours	19.9	2.1			16.8	0		
120 Hours	5.2	>0			13.4	0		
144 Hours	2.5	>0			10.2	0		
168 Hours	1.4	2.9			6.2	0.7		
Floaters	1.4	0			20.4	0		

^aData in the table are averages of duplicate determinations and are percentages based on oven-dry weights.

^bAWR = Accumulated Wood Recovery from the start of the flotation to the time indicated.

^cBCF is simply a calculation of % bark by oven-dry weight included with the recovered wood from a supposed original mixture of 75% wood and 25% bark.

^dBecause of the difficulties in adjusting the bark moisture content, the intended as well as the actual moisture contents are given in this table.

BCF of only 0.3% encouraged the use of the steam pressure cooker which is considered to be a device accelerating the liquid flotation segregation process.

Medium-sized ("on 1/2-inch") chips were used to make preliminary checks of modifications thought feasible. The charges used were equivalent to 250 grams oven-dry weight and were representative mixtures of corewood, slabwood, and bark of the sample chips. The first modification tried was the introduction of steam pressure using a procedure identical to that used for loblolly pine. See Fig. 1. Both 45% and 20% moisture content wood-bark chips were tried by placing them in an 85°C. water bath in the pressure cooker using 10 p.s.i. direct steam pressure and a treatment time of 5 minutes. After the pressure treatment the chips were skimmed from the hot bath and quenched in 22°C. water for 5 minutes, at which time the sinkers and floaters were labelled and the oven-dry weights determined.

The results (Table VII) showed no satisfactory segregation possibilities. The chips of 45% moisture content had 60% of the wood floating and 76% of the bark sinking. In the 20% moisture content chips 83% of the wood floated and only 25% of the bark sank.

In an examination of the bark chips left floating, it was noticed a resinous glaze seemed to seal the bark, particularly those particles originally at 20% moisture content. Since the bark chips were spongy, it was felt that compressing the chips and releasing them in water might break the glaze and cause more water absorption by bark chips. Several different compression-flotation tests were then performed.

Chips of 20% moisture content were processed by a water (22°C.), 5-minute agitated flotation. This flotation was followed by a double pass through the glue spreader rollers and a continued flotation on 22°C. water with the sunken

chips taken off at various intervals. The results, listed in Table VIII, indicate that from an original sample of 25% bark and 75% wood, 97% wood recovery (floating) was possible with a 1.7% BCF at 30 hours.

TABLE VII
WHITE SPRUCE
MODIFIED FLOTATION TEST
WITH
PRESSURE COOKER^a
ON 1/2-INCH WOOD-BARK CHIPS

	20% Moisture Content		45% Moisture Content	
	Wood	Bark	Wood	Bark
Floating chips, %	83.4	75.0	60.2	23.5
Sinking chips, %	16.6	25.0	39.8	76.5

^aBased on duplicate determinations of flotation tests using the pressure cooker. Conditions: 85°C. water bath, in cooker 8 minutes with 5 minutes at 10 p.s.i., chips quenched in 22°C. water after pressure cooker treatment where a 5-minute flotation determined sinking and floating chips. Percentages based on oven-dry weight of material (bark or wood). Data are percentages based on oven-dry weights of the material.

It was felt the use of chips with more than 20% but less than 45% moisture content might hasten water absorption and the sinking of the bark. A test was made of medium moisture content chips (35%) floated in 50°C. agitated water for 10 minutes, fed with 50°C. water through the glue spreader rollers twice and transferred to 22°C. water where the sinkers were extracted at periodic intervals up to 30 hours. The results (Table VIII) were similar to those of the previous compression flotation tests with a high percentage of bark sinking by the end of the 30-hour test and over 90% of the wood floating at 48 hours.

TABLE VIII
WHITE SPRUCE
MODIFIED FLOTATION TESTS
WITH
COMPRESSION
ON 1/2-INCH WOOD-BARK CHIPS^a

	Cool Water ^b Compression 20% Moisture Content Chips				Warm Water ^c Compression 35% Moisture Content Chips			
	Wood, %	Bark, %	AWR ^d Floaters, %	BCF, ^e %	Wood, %	Bark, %	AWR ^d Floaters, %	BCF, ^e %
Chips sinking:								
After first flotation	<0.1	4.3			0.8	8.0		
Compression								
1/4 Hour	0.2	16.4			NC	NC		
1/2 Hour	NC	NC			0.6	7.9		
1 Hour	NC	NC			<0.1	0.1		
1-1/4 Hour	0.1	9.9			NC	NC		
4 Hours	NC	NC			<0.1	7.9		
5 Hours	<0.1	14.5			NC	NC		
6 Hours	NC	NC			0.0	6.4		
8 Hours	0.4	7.6			NC	NC		
24 Hours	2.8	36.0			1.6	50.6	98.5	6.3
30 Hours	0.4	6.2	96.4	3.8	0.3	12.1	96.9	2.3
48 Hours	--	--	--	--	2.8	3.7		
Floaters	96.0	5.1	96.0	1.7	93.8	3.3		

^aValues given are percentages based on oven-dry weights of single determinations.
NC = No collection made.

^bConditions of test: 22°C. water all the way through compression. Through steel rollers twice.

^cConditions of test: 50°C. water until after compression, then 22°C. water.
Compression through steel rollers twice.

^dAWR = Accumulated Wood Recovery; wood recovered from the start of the flotation to the time indicated.

^eBCF is simply a calculation of % bark by oven-dry weight included with the recovered wood from a supposed original mixture of 75% wood and 25% bark.

The system used recovered 98.5% wood with a computed BCF (assuming 25% bark originally) of 6.3% at 24 hours and 96.9% wood with a 2.3% BCF at 30 hours. The results were little different from the previous test with the 20% moisture content chips. The chips apparently were not influenced by the water temperature. These preliminary tests, while not revealing a satisfactory white spruce wood-bark segregation system, because of the long dwell time necessary, do show conditions where segregation does take place in a satisfactory manner and a range of moisture contents within which the procedure works.

Because of time and budget limitations the preliminary modification investigations were terminated at this point and tests with pure fractions run. The tests involved the use of conditions which would require the least amount of modification to fresh chips and still give successful segregation. Pure fractions of 35% moisture content chips were processed in ambient water (about 22°C.) with a compression sequence at 10 minutes and at 4 hours. Corewood was kept separate from mature wood for this test sequence. The results of the pure fraction tests are listed in Table IX and pictured in Fig. 4.

In general, by using this flotation system, the wood chips still tended to float while the bark chips sank at an accelerated rate. The system did not allow an adequate differential to provide a "satisfactory" segregation of wood from bark chips.

The actual moisture content for the bark chips was again high at 42%, 7% above that desired. Because of this the data are slightly optimistic. The first point at which sunken chips were recovered occurred after ten minutes of agitated flotation and before any compressions were done. Percentage of bark sinking at this point for both sizes of chips was greater than had taken place

TABLE IX
WHITE SPRUCE
35% MOISTURE CONTENT^a

Sunken Chips Taken Off At:	On 1/4-Inch Chips					On 3/4-Inch Chips				
	Sinkers, %			AWR ^b	BCF, ^c	Sinkers, %			AWR ^b	BCF, ^c
	Wood		Bark			Wood		Bark		
	Core	Mature		Floaters, %	%	Core	Mature		Floaters, %	%
10 Min.	0.4	3.6	29.1			0.2	0	9.4		
1st Compres- sion &										
30 Min.	1.2	2.6	22.7			<0.1	0.3	10.1		
1 Hour	0.2	<0.1	1.0			<0.1	0	2.6		
2 Hours	0.2	<0.1	4.9			<0.1	0	3.0		
2nd Compres- sion &										
4 Hours	2.0	6.1	16.0	90.0	8.9	0.4	1.6	16.8		
6 Hours	<0.1	0.8	3.0	89.4	8.0	0	0	7.3	98.5	14.7
24 Hours	12.4	23.6	15.9	69.0	3.5	3.0	4.6	37.8	94.3	4.5
30 Hours	2.6	3.9	0.6			1.4	0.8	4.2	93.3	3.1
Floating chips	81.0	59.6	6.8			95.0	92.7	9.0		

^aData in the table are averages of duplicate determinations and are percentages based on oven-dry weights. Corewood is considered to be the first eight annual rings and mature wood is rings nine and beyond. Accumulated wood recovery is 28% of the corewood (the percentage of corewood in the original sample) and 72% of the mature wood.

^bAWR = Accumulated Wood Recovery from the start of the flotation to the time indicated.

^cBCF is simply a calculation of % bark by oven-dry weight included with the recovered wood from a supposed original mixture of 75% wood and 25% bark.

at 6 hours in the basic flotation test of 45% moisture content chips. Since these were the last tests performed, the chips had been stored at less than 40°F. for four months and storage is a factor unwittingly included in this test. While the extra five minutes of agitated flotation may well have increased bark sinking, it could not have influenced results that greatly. Therefore, both chip storage and extended agitated flotation were considered as factors contributing to the accelerated sinking of the bark.

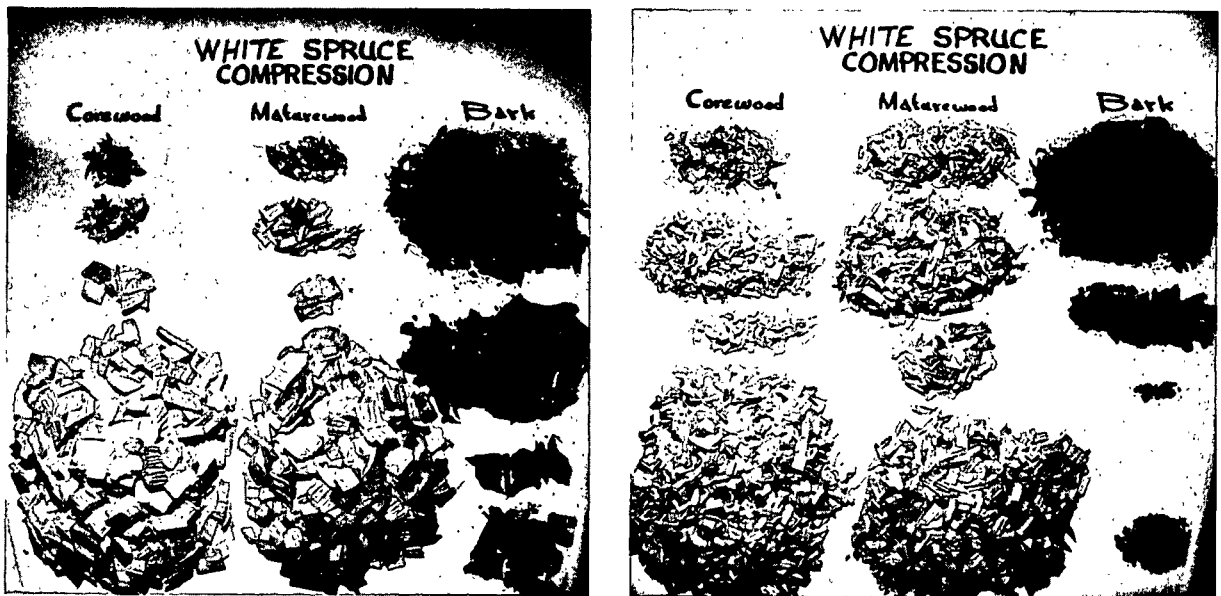


Figure 4. Results of the Pure Fraction White Spruce Modified Flotation Segregation Work (Large Chips in Left and Small Chips on Right). From Top to Bottom Are the Sinkers for First 4 Hours, From 4 Hours to 24 Hours, and From 24 Hours to 30 Hours and at the Bottom are the 30 Hour Floaters.

The compressions added at 10 minutes and 3-1/2 hours contributed to the accelerated sinking of the bark. Additions of two or more compressions in between the above times should further increase the amount of bark sinking. The use of 35% moisture content chips may have adversely influenced the segregation by causing the wood chips to sink earlier than is best for a good segregation. With this in mind, further work with this system using more compressions should also take into consideration the use of drier chips.

It was noted during the compression of the bark that many bark particles were squashed and stuck to the steel rolls and had to be doctored off. This seems to indicate that a compression segregation system, such as the Hosmer process [Blanchard (1)], might be a more appropriate segregation system for white spruce than a liquid flotation system. It is felt, however, that a liquid flotation segregation system for white spruce can be developed but that the system would require some rather exacting and/or complicated procedures.

Part of the answer may lie with the trees and the way they are chipped. The trees used for this study had only 6.7% bark and when chipped 78% of the bark was found in the large chips. If the above trends hold true for white spruce, i.e., trees with a low percentage of bark and the concentration of this bark going into one chip size class several possible solutions suggest themselves.

PLANS

Work in the final quarter of this project will be carried out on western larch and Douglas-fir. The revised basic flotation test will be used and emphasized will be the effect of chip size, chip moisture content, wood and bark characteristics, and dwell time on wood-bark chip segregation. If necessary, modified flotation sequences will be tested, among which will most surely be the pressure cooker treatments. Time permitting, a discussion of all the species will be given with emphasis on compatibility and problems with the various species.

ACKNOWLEDGMENTS

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LITERATURE CITED

1. Blanchard, F. G., U.S. pat. 3,070,318(Dec. 25, 1962).
2. Cole, D. E., Zobel, B. J., and Roberds, J. H. Slash, loblolly and longleaf pine in a mixed natural stand; a comparison of their wood properties, pulp yields, and paper properties. Tappi 49, no. 4:161-6(1966).
3. Gilmore, A. R., Metcalf, G. E., and Boggess, W. R. Specific gravity of short-leaf pine and loblolly pine in southern Illinois. J. Forestry 59, no. 12:894-96(1961).
4. Hale, J. D. Thickness and density of bark; trends of variation for six pulpwood species. Pulp Paper Mag. Can. 56, no. 13:113-17(1955).
5. Isenberg, I. H. Pulpwoods of the United States and Canada. 2nd ed. Appleton, Wis., The Institute of Paper Chemistry, 1951. 187 p.
6. Martin, R. E. Characterization of southern pine barks. Forest Prod. J. 19, no. 8:23-30(1969).
7. Martin, R. E., and Crist, J. B. Selected physical-mechanical properties of eastern tree barks. Forest Prod. J. 18, no. 11:54-60(1968).
8. Peck, E. C. Specific gravity and related properties of softwood lumber. U.S.D.A. Tech. Bull. no. 343, 1933.
9. U.S. Forest Products Laboratory. Wood handbook. U.S.D.A. Agriculture Handbook 72, 1955.
10. Wahlgren, H. E., Baker, G., Maeglin, R. R., and Hart, A. C. Survey of specific gravity of eight Maine conifers. U.S.D.A., Forest Service Research Paper FPL 95, 1968.
11. Zobel, B. J., and McElwee, R. L. Natural variation in wood specific gravity of loblolly pine, and an analysis of contributing factors. Tappi 41, no. 4:158-61(1958).
12. Zobel, B. J., and Rhodes, R. Specific gravity estimations of mature loblolly pine from juvenile wood and seedling limb sections. Forest Sci. 2:107-12 (1956).

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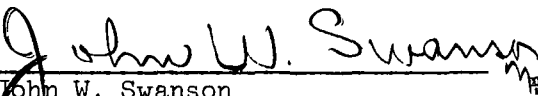
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APPENDIX

TABLE X

WOOD AND BARK SPECIFIC GRAVITIES AND REFERENCES

Source	Loblolly Pine				Shagbark Hickory Wood	White Spruce	
	Wood			Bark Outer		Wood Total	Bark Total
	Core	Mature	Total				
Cole, <u>et al.</u> (<u>2</u>)	0.40-0.50	0.47-0.55	0.46-0.62				
Gilmore, <u>et al.</u> (<u>3</u>)			0.36-0.47				
Hale (<u>4</u>)						0.35	0.32-0.37
Isenberg (<u>5</u>)			0.47				
Martin (<u>6</u>)				0.47			
Martin & Crist (<u>7</u>)				0.29-0.51			
Peck (<u>8</u>)			0.47				
U.S. Forest Products Lab. (<u>9</u>)			0.47		0.35	0.37	
Wahlgren (<u>10</u>)							0.35-0.37
Zobel & McElwee (<u>11</u>)	0.35-0.48	0.40-0.68	0.43-0.62				
Zobel & Rhodes (<u>12</u>)	0.52-0.53		0.54-0.55				